

Navy Case No. 79,693

In the United States Patent and Trademark Office

In re: Bayya et al
Serial No.: 09/699,396
Filed: October 31, 2000
For: Method For Coating
Small Particles

Examiner: Michael B. Cleveland
Art Unit: 1762
Date: July 24, 2003

Revised Appeal Brief

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20230:

Sir:

This is an appeal from the final rejection of claims 1,3-8, 10-13 and 15-20. No claims have been allowed.

(1) Real party in interest

The real party in interest in this patent application is the Federal Government, as represented by the Department of Navy.

(2) Related appeals and interferences

This application is not involved in any other appeal or interference.

(3) Status of claims

The claims on appeal are the finally rejected claims 1,3-8, 10-13 and 13-20.. Claims 2 and 9 were canceled by the amendment dated August 14, 2002, and claim 14 is to be canceled by the accompanying amendment dated April 4, 2003.

(4) Status of amendments

The amendment dated Feb. 6, 2003, and entitled "Amendment After Final Rejection" has not been entered into the record,

however, the accompanying amendment dated April 4, 2003, is expected to be entered, based on the Examiner's comments and recommendations in the Advisory Action dated Feb. 28, 2003. This Appeal Brief has been prepared on the assumption that the Second Amendment After Final Rejection, dated April 4, 2003, will be entered.

(5) Summary of the invention

As disclosed at the bottom half of p.9 of the specification identified above, the coating method of this invention includes the steps of dissolving a coating precursor in a solvent to form a precursor solution; optionally adding a miscible diluent to the solution to form a coating solution; adding solid particles to the coating or the precursor solution to form a coating slurry which need not contain the diluent, with the particles surrounded with the coating or the precursor solution; adjusting temperature, pH and/or another parameter to discourage precipitation on the particles; delivering the slurry to an atomizer without separating the particles from the coating or the precursor solution; atomizing or spraying the slurry in the form of droplets through a drying zone to remove volatile matter from the particles/droplets, with the droplets containing at least one particle; heat-treating the coated particles/droplets; and collecting the heat-treated coated particles/droplets. The method can commence with the step of adding the particles to a coating or precursor solution instead of initially dissolving the coating precursor in the solvent to from the precursor solution.

Furthermore, the adjusting step can be dispensed with by providing at end of the adding step in section (a) of claim 1 that the precursor is not precipitated until after the spraying step.

The essence of the invention resides in delaying precipitation of the coating material on the particles until after the spraying step. This difference allows the coated phosphor particles used in field emission displays to unexpectedly meet the standard of continually operating for in excess of 10,000 hours without losing 50% of its original brightness, which the standard was not met prior to this invention, as noted at bottom of p.5 of this specification. The prior art phosphor particles have lifetime of only 2,000-3,000 hours.

(6) Issues

- (a) Whether claims 1, 3-8 and 11-18 are enabled under the first paragraph of 35 USC 112;
- (b) Whether claims 1,3 and 13 are obvious under 35USC 103(a) over the Petersen reference in view of the Strom, Anderson and Okabe references;
- (c) Whether claims 3-8,12 and 15-19 are obvious under 35USC 103(a) over the Petersen reference in view of the Strom, Anderson and Okabe references, as applied to claims 1 and 13, and further in view of the Masters reference;
- (d) Whether claims 10 and 20 are obvious under 35USC103(a) over the Petersen, Strom, Anderson, Okabe, and Masters

references, as applied to claims 8 and 13, and further in view of the Hanneman and Chau references;

(g) Whether claim 11 is obvious under 35USC103(a) over the Petersen, Strom, Anderson, Okabe, Masters, Hanneman, and Chau references, as applied to claim 10, and further in view of the Ohoshi reference.

(8) Arguments

(I) This is in reference to the rejection of claims 1, 3-8 and 11-19 on the first paragraph of 35 USC 112. In the Final Rejection dated Nov. 21, 2002, the Examiner stated, inter alia, that the specification does not provide enablement for the formation of organic coatings.

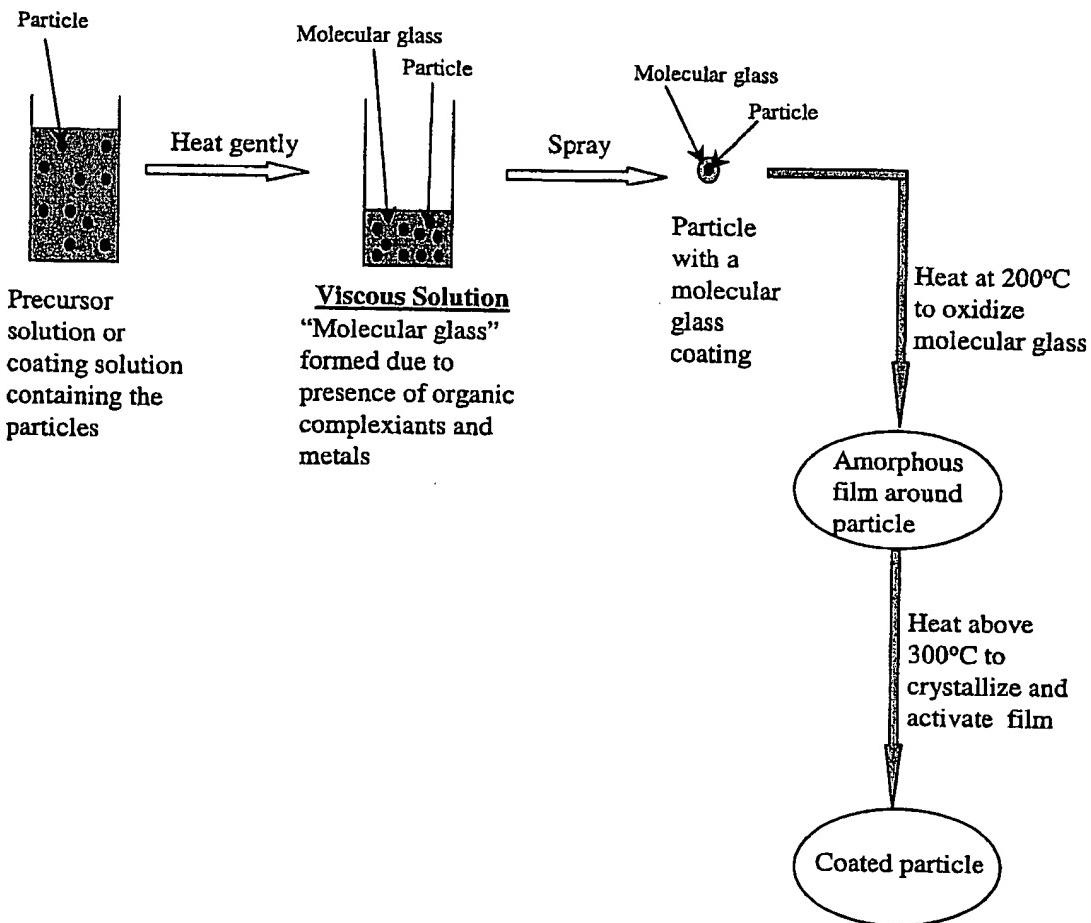
It was believed that the rejection of claims 1, 3-8 and on the first paragraph of 35 USC 112 has been resolved since the precursor indium methyl (trimethyl) acetyl acetate, claimed in claim 8, is an organic precursor which has been originally disclosed and claimed herein.

(IV) All of the art rejections appealed from are on 35USC103(a) and pertain to obviousness of the subject matter claimed in claims 1,3-8, 10-13 and 15-20. All of the appealed claims contain the limitation "the precursor is not precipitated until after spraying" which is responsible for the unexpected result of a particle lifetime in excess of 10,000 hours of continuous operation without losing 50% of the original brightness. The coated particles are phosphor particles, as recited, for instance, in claims 11 and 18, that can be used in

field emission displays. None of the applied references neither discloses nor suggests delaying precipitation in order to attain the unexpected result.

All of the obviousness rejections rely on the Petersen reference, in combination with other references, to reject as obvious the subject matter claimed in the appealed claims. The Petersen reference does not disclose anything that would render the herein-claimed method obvious, alone or in combination with any of the other applied references. A side-by-side comparative color chart of the Petersen reference method and the herein-claimed Naval Research Laboratory (NRL) method, given to the Examiner during the interview on July 30, 2002, and attached hereto and marked "Exhibit 1NC79693" shows formation of the coating material on the particle after the spraying step in the NRL method and formation of the coating material on the particle prior to the spraying step which difference results in unexpected advantages. Independent claims 1 and 13 were amended to focus on this difference by reciting that the precursor was not precipitated until after the spraying step. Furthermore, it is believed that the Petersen reference leads away from the herein-claimed method by causing precipitation before the spraying step. The case of In re Wagner, 152 USPQ 559 (1967), and its progeny, stands for the proposition that prior art that leads away from the invention does not render the invention obvious.

In reference to paragraph, above, the Examiner should consider the following sketch of Peterson's method and comments



that follow;

Peterson has to add organic complexants (e.g. urea and carbohydrazide) to form a molecular glass prior to spraying. Obviously, he does not have a solution at this point prior to spraying but instead, a molecular glass. A molecular glass surrounding the particles is not formed prior to or after spraying. Claim 1 herein leads away from making a molecular glass. After spraying, the Peterson reference creates a particle

coated with molecular glass. This is not done here, but instead, a particle coated with the precursor or coating solution is sprayed.

Furthermore, none of the 8 applied reference discloses or suggest any combination of references that would render obvious the delay of precipitation. See the case of Lndemann Maschinenfabrik v. American Hoist and Derick Co., 221 USPQ 481, and its progeny, which stands for the proposition that something in the prior art as a whole must suggest the desirability, and thus the obviousness, of making the combination. None of the applied references, singly or in combination, disclose or suggest to delay precipitation in order to prolong lifetime and brightness of the coated particles.

The Examiner herein used hindsight knowledge of this invention to reject the calims on various combinations of references. The case of Interconnect Planning Corp., 227 USPQ at 551, and its progeny, prohibits the use of hindsight knowledge in the rejection.

8(a). Claims 1,3 and 13 were rejected as being obvious over the Petersen reference in view of the Strom, Anderson and okabe references. The Examiner has concluded that it would have been obvious to prevent any gellation or precipitation in Petersen's slurry before spray drying in order to avoid destroying flow properties of the solution so that a more uniform coating could be made.

The Petersen reference pertains to phosphor particles and to a method for making same, the Strom reference pertains to films, the Anderson reference pertains to alumina, and the Okabe reference pertains to ceramic powder. As noted in the case of In re Francis Flick and Bower, 101 USPQ 70, and adhered to by its progeny, the test as to whether references are from non-analogous arts is whether one seeking to solve a problem with respect to one art be apt to seek the solution to the problem in the other art. It is submitted that, at least some of the applied references, are from non-analogous arts and are not combinable since one skilled in the art of coating particles would not seek solution to a problem in at least some of the secondary references.

8(b). Claims 3-8, 12 and 15-19 were rejected as being obvious over the Petersen reference in view of the Masters reference. The Masters reference is a handbook on spray drying and its technology is older than 1987. There is nothing in the Petersen or the Masters references that teaches or suggests delaying precipitation for any reason. Furthermore, to avail oneself of delayed precipitation, one has to take appropriate steps prior to the spray drying step, which leads one to question as to why the Masters reference was combined with the Petersen reference.

8(c). Claims 3-8, 12 and 15-19 were rejected as being obvious over the Petersen, Strom, Anderson, and Okabe references, as applied to claims 1 and 13, and further in view of the Masters

reference. The reasons why these claims are unobvious over the applied references appear in paragraphs 8(a) and 8(b), above.

8(d). Claims 10 and 20 were rejected as being obvious over the Petersen reference in view of the Masters reference, as applied to claims 8 and 13, and further in view of the Hanneman and Chau references. Unobviousness in connection with claims 8 and 13 on the Petersen and Masters references was argued in paragraph 8(b), above, and those arguments apply here as well. The Hanneman reference pertains to coating materials but has nothing to do with delaying precipitation and the Chau reference pertains to phosphor particles but it also has nothing to do with delaying precipitation. Furthermore, the Examiner has not used the Hanneman and Chau references to show delayed precipitation but to show obviousness of forming an ethyl silica diffusion barrier coating.

8(e). Claims 10 and 20 were rejected as being obvious over the Petersen, Strom, Anderson, Okabe, and Masters references, as applied to claims 1 and 13, and further in view of the Hanneman and Chau references. Unobviousness of claims 1 and 13 on the Petersen reference in view of the Strom, Anderson, and Masters references was argued in paragraphs 8(a) and 8(b), above, and apply here as well. Arguments re the Hanneman and the Chau references appear in paragraph 8(d), above, and apply here as well.

8(f). Claim 11 was rejected as being obvious over the Petersen, Masters, Hanneman, and Chau references, as applied to

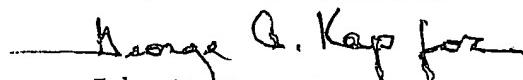
claim 10, and further in view of the Ohoshi reference. Unobviousness of claim 10 was argued in paragraph 8(a), above, and arguments apply here as well. The Ohoshi reference pertains to a light-emitting device and method of making it. The Ohoshi reference is not analogous to coating of particles and is improperly combined with the other references. Also, the Examiner is using the Ohoshi reference to show obviousness of using the ZnS:Ag,Cl particle for field effect devices and not delay of precipitation. The Ohoshi reference does not disclose nor suggest delaying precipitation of a coating material on a particle.

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The Examiner contends that clean copy and marked-up copy of claim 1 of the After Final amendment do not agree with each other. The Examiner should select whatever claim he wishes for appeal purposes, subject to the acquiescence of the undersigned.

Enclosed also is the Third Amendment After Final Refection.

Respectfully submitted


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CERTIFICATE OF FACSIMILE TRANSMISSION

I hereby certify that this document is faxed to the PTO on the date shown below in triplicate.

July 24, 2003

Date

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(9) Appendix

Claims on appeal are the following claims 1,3-8,10-13 and 15-20:

1. A method for coating solid particles comprising the steps of
 - (a) adding solid particles to a liquid coating solution or precursor solution to form a liquid coating slurry containing a coating precursor, solvent for the precursor and the solid particles dispersed therein whereby the precursor material is not precipitated until after spraying,
 - (b) spraying the coating slurry to form droplets containing at least one particle,
 - (c) passing the droplets through a zone where the droplets are dried and form dry coated particles wherein the coating material is formed from the coating solution or the precursor material, and
 - (d) heat treating the coating material on the particles to remove volatile matter from the coating material.

3. The method of claim 1 wherein temperature in the zone is elevated and the heat treatment of the coated particles is conducted at a temperature above the elevated temperature in the zone, and the precursor is selected from the group consisting of alkoxides, nitrates, sulfates, acetates, hydroxides, hydrates, chlorides, other precursors that can be dissolved in aqueous or non-aqueous liquids, and mixtures thereof.

4. The method of claim 3 wherein the particles are less than about 100 microns in diameter, dilution ratio in the coating slurry of milliliters of coating solution or precursor solution per gram of phosphor particles is 100-5000, thickness of the coating material on the particles is 1-1000 nm, velocity of the droplets in the zone is 0.1-1000 cm/sec and residence of the droplets in the zone is from instantaneous to a fraction of a minute.

5. The method of claim 3 wherein the particles are less than about 50 microns in diameter, temperature in the zone is 100-500°C, dilution ratio in the coating slurry of milliliters of coating solution or precursor solution per gram of phosphor particles is 200-3000, thickness of the coating material on the particles is 2-200 nm, velocity of the droplets in the zone is 1-50

cm/sec, and residence time of the droplets in the zone is 0.1-10 seconds.

6. The method of claim 5 including the step of mixing a precursor solution with a diluent which diluent is miscible with the precursor solution to form the coating solution, the precursor solution containing coating precursor during droplet formation.

7. The method of claim 6 wherein the residence time of the droplets through the zone is 1-5 seconds, the particles are phosphor particles, and said heat treating step is carried out at 50-2000°C over a period of 0.01-48 hours.

8. The method of claim 7 wherein said heat-treating step is carried out at 300-1500°C over a period of 0.1-24 hours.

10. The method of claim 8 wherein the coating material is selected from the group consisting of indium tin oxide, silicon dioxide, magnesium oxide, sodium phosphate, yttrium-europium oxide, and mixtures thereof; and the precursor is selected from the group consisting of indium methyl (trimethyl) acetyl acetate, tin isopropoxide, tetraethyl orthosilicate, magnesium nitrate, yttrium chloride, europium chloride, sodium phosphate and mixtures thereof.

11. The method of claim 10 wherein the particles are ZnS:Ag:Cl phosphor particles.

12. The method of claim 4 including the step of providing at least one more coating on the coated particles.

13. A method comprising the steps of

- (a) preparing a liquid precursor solution by dissolving a coating precursor in a liquid precursor solvent;
- (b) mixing the precursor solution with a diluent, that is miscible with the precursor solvent, to form a liquid coating solution;
- (c) adding with mixing solid particles to the coating solution to form a liquid coating slurry containing the coating precursor dissolved in the coating solution and the solid particles dispersed therein whereby the precursor is not precipitated until after spraying;
- (d) spraying the coating slurry to form droplets containing at least one

particle;

(e) passing the droplets through a zone where the droplets are dried and form dry particles coated with a coating material formed from the precursor solution;

(f) heat-treating the coating material on the particles to remove volatile matter on the coating material and to convert the coating material from electrically non-conducting amorphous to electrically conducting crystalline.

15. The method of claim 13 wherein temperature in the zone is elevated and the heat treatment of the coated particles is conducted at a temperature above the elevated temperature in the zone, and the precursor(s) is selected from the group consisting of alkoxides, nitrates, sulfates, acetates, hydroxides, hydrides, chlorides, other precursors that can be dissolved in aqueous or non-aqueous liquids and mixtures thereof.

16. The method of claim 15 wherein the particles are less than about 100 microns in diameter, dilution ratio in the coating slurry of milliliters of coating solution or precursor solution per gram of phosphor particles is 100-5000, thickness of the coating material on the particles is 1-1000 nm, velocity of the droplets in the zone is 0.1-100 cm/sec and residence of the droplets in the zone is from instantaneous to a fraction of a minute.

17. The method of claim 16 wherein the particles are less than about 50 microns in diameter, temperature in the zone is 100-500°C, dilution ratio in the coating slurry of milliliters of coating solution or precursor solution per gram of phosphor particles is 200-3000, thickness of the coating material on the particles is 2-200 nm, velocity of the droplets in the zone is 1-50 cm/sec, and residence time of the droplets in the zone is 0.1-10 seconds.

18. The method of claim 17 wherein the residence time of the droplets through the zone is 0.1-10 seconds, the particles are phosphor particles, and said heat treating step is carried out at 200-2000°C over a period of 0.01-48 hours.

19. The method of claim 17 wherein said heat-treating step is carried out at 300-1500°C over a period of 0.1-24 hours and wherein the coating

material is selected from the group consisting of inorganic, organic and inorganic/organic hybrids.

20. The method of claim 13 wherein said heat-treating step is carried out at 300-1500°C over a period of 0.1-24 hours and wherein the coating material is selected from the group consisting of indium tin oxide, silicon dioxide, magnesium oxide, sodium phosphate, yttrium-europium oxide, and mixtures thereof; and the precursor solution includes a precursor selected from the group consisting of indium methyl (trimethyl) acetyl acetate, tin isopropoxide, tetraethyl orthosilicate, magnesium nitrate, yttrium chloride, europium chloride, sodium phosphate and mixtures thereof.

Petersen, Chau Method

NRL Method

Dissolve precursors
in solvent

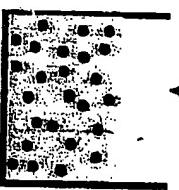
Add particles

Dissolve precursors
in solvent

Add particles

Cause deposition of coating
while in the beaker

separate coated particles from liquid
e.g. spraying, filtration, boiling



Spray, no deposition

Cause deposition of coating
by the removal of liquid in
the sprayer

